SOLID-PHASE EXTRACTION WITH CARTRIDGES

D. A. Wells, Sample Prep Solutions Company, Maplewood, MN, USA

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Introduction

Sample preparation is an important component of an analytical method. It is used to concentrate an analyte to improve its limits of detection, as well as to isolate an analyte from unwanted matrix components that can cause interferences upon analysis. Solid-phase extraction (SPE), as a tool for this sample concentration and isolation, has gained acceptance since its commercial introduction circa two decades ago. SPE is performed using commercial packed cartridges (containing approximately 50–500 mg packing material) as well as discs (containing from 4–500 mg). Many formats, chemistries and sizes of SPE products are available to meet a range of separation needs.

Solid-phase extraction is preferred to other types of sample preparation techniques, such as liquid–liquid extraction (LLE), for many reasons. SPE is an efficient technique, often achieving higher recovery of analyte than other methods of sample preparation because of its selectivity. The chemistry of attraction between an analyte and the solid sorbent can be exploited by pH and solvent considerations to allow interaction yet exclude interferences. SPE is a less time-consuming and labour-intensive technique. Extraction typically involves adding different liquids through SPE columns in parallel and collecting the eluate at the final step. Emulsion formation is eliminated – in LLE an emulsion sometimes forms between the aqueous and organic layers preventing phase separation. Organic solvent consumption is far less using SPE than typical LLE techniques, saving money in terms of both purchase costs of solvents and costs to dispose of these regulated solvents. Reduced exposure of the analyst to organic solvents also improves safety in the laboratory. Unlike LLE the SPE procedure using columns can be automated. There are several hardware choices available commercially that transform SPE from a manual procedure into a fully automated one, allowing the analyst to perform other tasks in the laboratory. Batch procedures of automation are available in which a number of samples are extracted to yield the same number of eluates ready for analysis. On-line serial automation is common, in which a sample is extracted then injected by the instrument and, while analysis is ongoing, the next sample is extracted. Miniaturization of SPE allows the convenient use of smaller sample sizes and the ability to physically work with eluates as small as 50 μL when using the disc format.

The SPE technique has been shown to be useful for a variety of sample matrices, including (but not limited to): drinking water and river water, air, biological fluids (e.g., blood, serum, plasma, urine), tissues, peptides, drug formulations, microbial broths, animal feed, beverages, fruits and vegetables, and soil. Thus, the numbeBr of applications for this sample preparation technique in the literature is extensive and can be found spanning the last 25 years. It is the goal of this chapter to highlight many of the applications of SPE for a variety of sample matrices and demonstrate the versatility and usefulness of this sample preparation technique (**Table 1**).

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Table 1 Examples of classical analytical applications using solid-phase extraction

Market	*Application*
Environmental	Trace enrichment of organic pollutants from water
	Organic acids, detergents and surfactants from water
	Insecticides and pesticides from soil
	Explosives residues in groundwater
	Oil and grease analysis
Food	Pesticides in fruits and vegetables
	Sodium benzoate in colas and fruit juice
	Plant growth regulators in spinach juice
	Toxic fungal metabolites in rodent feed
	Vitamins in food
	Cholesterol oxidation products in milkfat
	Caffeine in beverages
	β-Agonists and antibiotics in meat products
Biotechnology	Purification and fractionation of proteins and peptides
	Desalting of peptides
	Purification of DNA from microbial broths
Pharmaceutical	Antibiotic content in ointments
	Aspirin content in tablets
	Drugs in serum, plasma and urine
Clinical	Catecholamines in plasma and urine
	Lipids in serum
	Drugs in tissues
	Vitamins and steroids in serum
	Cyclosporin in blood

Environmental Applications

Trace Enrichment from Environmental Samples

The gas and liquid chromatographic analyses of polar pollutants in waters (e.g. for drinking, river and effluent) require a concentration step before analysis to determine part-per-million levels and lower, as regulations specify. Solid-phase extraction is the most widely used technique for trace enrichment of polar environmental pollutants since it uses low volumes of hazardous organic solvent, can be automated using cartridges and discs, and the analysis can be done either off-line in batches or on-line with the chromatographic system.

Typically, 1 L volumes of water samples are required for analysis in the United States, as mandated by the Environmental Protection Agency (EPA). Hydrophobic C18 and C8 sorbents are commonly used for the majority of trace enrichment needs; the analyte structure dictates the optimal sorbent chemistry. Practical considerations for passing 1 L of water through a SPE cartridge favour the use of larger diameter (47 or 90 mm) discs (e.g. glass fibre and PTFE-based) for these applications. While both discs and cartridges can be automated, discs are preferred for their much larger cross-sectional surface area. Using discs, liquid can be passed through at high flow rates without loss of analyte, thus reducing the extraction time to about 10–15 min instead of about 1 h for narrower cartridges.

Aqueous samples (100 mL to 1 L) containing organochlorine pesticides (e.g. lindane, methoxychlor, or endosulfan) can be concentrated from water, made acidic by using C18 bonded silica in 47 mm discs. Elution from the sorbent is accomplished using 3–5 mL aliquots of ethyl acetate, from which water is removed in a separate step using anhydrous sodium sulfate, it is then concentrated before analysis using gas chromatography (GC) with electron capture detection (ECD). Polyaromatic hydrocarbons (e.g. phenanthrene, pyrene, anthracene), organophosphorous pesticides (e.g. diazinon, methyl parathion), and herbicides (e.g. atrazine, alachlor) may be analysed in a similar manner using C18 sorbent and ethyl acetate elution from discs prior to GC analysis.

Phenols and chlorinated phenols are moderately polar compounds that can display ionic character at pH values above 7. Another group of polar compounds displaying ionic character is the acid herbicides (e.g. 2,4-dichlorophenoxyacetic acid, 2,4,5-trichlorophenoxyacetic acid and dicamba). Rather than C18 bonded silica, a more efficient sorbent for extraction of these types of compounds is polystyrene divinylbenzene (SDB), an organic polymer. SDB has a slightly different selectivity than C18, owing to its aromaticity, that allows it to extend its range of attraction to include more polar species such as phenols. Other advantages of SDB are that it is totally organic, is stable across the entire pH range, and has greater capacity per gram than comparable reversed-phase bonded silica sorbents. Extraction of these phenols is performed with a SDB disc (or cartridge). Elution from the sorbent is accomplished using 3–5 mL aliquots of acetonitrile (methanol or acetone may be substituted) before analysis.

Diquat and paraquat are examples of polar compounds that are quaternary amines, thus always positively charged. These analytes are found only in very small concentrations in water, since they more readily attract to soils and plants via their cationic functionality. They can be concentrated from water on a cyano sorbent or some types of C8 sorbent, those that have a high degree of residual silanols available for attraction of cationic species. The extraction method for C8 includes an ion-pairing agent, to which paraquat and diquat bind. Elution with 5 mL methanol containing acid and diethylamine disrupts this binding; analysis is by HPLC. **Table 2** lists examples of USA EPA methods employing disc solid-phase extraction.

On-Line Techniques

While SPE is a successful technique performed in batch mode before the analysis step, there can be drawbacks such as loss of sensitivity (only an aliquot of the total mass isolated is used), losses due to evaporation or during transfer and contamination from external sources. Instrumentation has now advanced to allow for on-line trace enrichment, where the sample eluent is injected onto a high-pressure liquid chromatography (HPLC) apparatus. The sample can be isolated on a guard column while the HPLC is running the previous sample, so time is not lost between isolation and analysis. The cartridge performing the extraction on-line, coupled to a liquid chromatographic system, can be commercially bought, such as the PROSPEKT system (Spark Holland) or constructed by hand using Empore® (3M Company) membrane extraction discs placed into a holder (4.6 mm internal diameter). Multi-residue methods that extract a variety of pesticides (acidic, neutral and basic) from waters are commonly used. In order to preconcentrate all these compounds simultaneously, it is necessary in most cases to acidify the sample and use a C18 bonded silica or polymer-based SDB sorbent in series with a cation exchanger. In order to avoid rapidly overloading the cation exchanger with samples of high ionic strength, calcium ions are first precipitated

Table 2 United States Environmental Protection Agency EPA methods allowing the use of solid-phase extraction for sample preparation

Method number	*Analytes*	*Sorbent*	*Analysis technique*[a]
506	Phthalate and adipate esters in drinking water	C18	GC/PID
507	Nitrogen and phosphorous containing pesticides in water	C18	GC/NPD
508	Chlorinated herbicides and organochlorine pesticides in water	C18	GC/ECD
513	TCDD (2,3,7,8-tetrachlorodibenzo-*p*-dioxin) in drinking water	C18	GC/MS
515.2	Chlorinated acids in water	SDB	GC/ECD
525.1	Organic compounds in drinking water	C18	GC/MS
548.1	Endothall	Strong anion exchange	GC/MS
549.1	Diquat and paraquat in drinking water	C18, C8 or strong cation exchange	HPLC/UV
550.1	Polycyclic aromatic hydrocarbons (PAH) in drinking water	C18	HPLC/UV and flurorescence
552.1	Haloacetic acids and dalapon in drinking water	Strong anion exchange	GC/ECD
553	Benzidines and nitrogen containing pesticides in water	C18	HPLC/MS
554	Ozonation disinfection by-products (carbonyl compounds)	C18	HPLC
1613 Revision B	Tetra- to octa-chlorinated dioxins and furans	C18	HRGC/HRMS
1664	Oil and grease	C18	Gravimetric and infrared
3535 (SW846)	Organochlorine pesticides and phthalate esters from groundwater, wastewater and TCLP leachates	C18, SDB	Various GC/ECD techniques

[a]Abbreviations: GC, gas chromatography; PID, photoionization detector; ECD, electron-capture detector; MS, mass spectrometry; HPLC, high-performance liquid chromatography; UV, ultraviolet, HRMS, high resolution mass spectrometry.

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with oxalic acid and heavy metals are complexed with ethylenediaminetetraacetic acid (EDTA) prior to SPE. SPE cartridges on-line are sometimes preloaded with sodium dodecyl sulfate (SDS) to improve retention of basic pollutants at low pH.

Organic Acids Found in the Environment

The trace determination of EDTA in environmental water samples is an example of an analytical challenge – one in which the analyte readily chelates with metals, is very water soluble and is an organic acid. EDTA is commonly used in the clean-up of radioactivity and heavy metal wastes, and is also found in the environment as a detergent and water softening agent. Chelation of EDTA with toxic metals facilitates the migration of these hazardous materials from ground dumps into a water-soluble state where they can be transported into lakes, rivers and streams. The sample preparation technique of choice for EDTA is SPE since it improves detection limits compared with other techniques and can be fully automated. The lowest detection limits (0.15 $\mu g\,L^{-1}$, five times lower than previously reported methods using GC-mass spectrometry (MS) and HPLC) have been obtained using capillary electrophoresis (CE) with ion-spray tandem mass spectrometry (MS-MS) for selective detection.

The sample preparation of EDTA from water samples (5 mL) involves conversion of all free and chelated EDTA present into the nickel EDTA chelate by adding 100 μL $Ni(NO_3)_2$ at a pH from 7–9. The pH is then adjusted to about 3.0 using about 12 μL of 9% formic acid. This sample is added to preconditioned strong anion exchange solid-phase extraction cartridges (SPEC® (Ansys Diagnostics glass fibre disc cartridges). Wash solvents used (in order) are water (adjusted to pH 3.0 with formic acid), water (neutral pH), and methanol. Finally, the NiEDTA is eluted using a solution of 50 mM trifluoroacetic acid, 1 mM bromothymol blue and 5% methanol. The eluate is evaporated to dryness, reconstituted in 0.1% ammonium hydroxide, evaporated to dryness again, and then reconstituted in 30 μL water for analysis. This extract is then analysed by CE-MS. The strongly acidic elution solvent dissociates the NiEDTA complex, while bromothymol blue displaces the remaining NiEDTA from the disc. Reconstitution in ammonium hydroxide facilitates the re-complexation of the NiEDTA.

Food Applications

Pesticides in Food

Food applications using SPE present complexities that are not encountered in water extractions. Substances such as apples, lettuce, tomatoes and strawberries have tissue components that must be removed before extraction, and the analytes within the tissue fluids must be made available for extraction or removal prior to analysis. Multiple pesticides are commonly analysed in food crops. One popular multiresidue screening technique is the Luke II method, in which a crop sample (100 g) is homogenized with a water-miscible solvent (acetone). However, other crop materials that have solubility in acetone are also extracted. The solvent and water from the crop are then filtered and the filtrate subjected to a series of liquid–liquid partitioning extractions. The resulting mixture is subjected to two or more SPE packed cartridge (or disc) clean-up steps using sorbents with varying selectivity to remove co-extracted materials while pesticides pass through. Use of SPE techniques within this method allows for reduced solvent use and improved throughput.

A variation of this approach described uses SPE discs with reversed-phase sorbents (SDB-RPS disc stacked on top of a carbon disc; 3M Company) to capture the pesticides, rather than the co-extracted substances that often use normal-phase sorbents. This disc procedure is as follows. A 100 g sample of each crop material is mechanically blended with 100 mL acetone. The puree is filtered through a glass fibre filter and three 10-mL aliquots (10 g crop equivalent, wet weight) of each filtrate are transferred to centrifuge tubes, and the volume is reduced under nitrogen to about 5 mL. Water is added to adjust volume to 15 mL. SPE discs are conditioned with acetone, followed (in order) by ethyl acetate, methanol, then water. Samples are filtered through each disc. When the entire sample has been extracted, the discs are removed and inverted, so that SDB-RPS is on the bottom and carbon on the top. Elution is accomplished with 2 mL acetone, followed by two successive 5-mL aliquots of ethyl acetate. The eluent is dried using anhydrous sodium sulfate, then concentrated by evaporation to 5 mL volume, and analysed by GC-ECD. The combination of SDB-RPS and carbon sorbent chemistries for the extraction is superior to reversed-phase bonded silica sorbents to extend the range of attraction to the more polar pesticides with high water solubility, namely dimethoate, *o*-methoate and methamidophos. SDB-RPS contains the SDB chemistry but because of the nature of sulfonic acid groups bonded on the SDB surface it captures cationic moieties also. Carbon is used to capture analytes not retaining on SDB-RPS. By reversing the order of sorbents for elution, the pesticides never come in contact with the carbon and are quantitatively recovered.

β-Agonists in Cattle Meat

β_2-Agonists (e.g. clenbuterol, brombuterol, mabuterol and mapenterol), originally developed for treatment of chronic obstructive pulmonary diseases in humans, have been misused as a repartitioning agent in the fattening of cattle. When cattle are treated, residues may remain in the meat and liver. In order to monitor regulatory bans on use of these drugs in cattle, samples are removed at slaughterhouses and analysed for the presence of these illegal growth promoters. Urine is the matrix most commonly used for the analysis of these β-agonist drugs. Solid-phase extraction has been shown to be an effective technique for these drugs, using reversed-phase or mixed-mode sorbents (containing both reversed phase and cation exchange functionalities). The SPE procedure adds 1 mL 0.5 M potassium phosphate buffer pH 4.0 to 5 mL urine, followed by centrifugation. A mixed-mode sorbent bed is conditioned with methanol, water, then 0.1 M potassium phosphate buffer pH 4.0. The sample is loaded onto the cartridge, followed by a wash solvent of 70% methanol in water. After drying the cartridge, elution is accomplished with ethanol–n-hexane–ammonium hydroxide (70 : 25 : 5, v/v/v) in two sequential portions. Solvent is evaporated under nitrogen and heat and reconstituted in 25% acetonitrile in water for HPLC analysis.

Biotechnology Applications

Purification and Fractionation of Proteins and Peptides

Proteins are significant components of most physiological samples. It is often important to measure very low concentrations of specific peptides in biological fluids for diagnosis of disease states and to investigate physiological roles of certain peptides. Examples include examining the role of atrial natriuretic peptide in cardiovascular disease, studying β-endorphins involved in the neurochemistry of the brain, and isolating lymphokines to monitor their effect on immune system regulation. The quantification of a peptide such as casein in milk products is an application in the food area requiring isolation and purification. Solid-phase extraction is commonly used as a preliminary purification step to remove cross-reacting or interfering materials in sample matrices before analysis.

One common approach to purifying hydrophilic proteins or peptides is to fractionate crude proteinaceous extracts and remove hydrophobic proteins. Proteins above 15–20 000 molecular weight are usually too large and cannot easily enter the pores of typical 60–100 Å bonded silica particles. Thus, these large proteins pass unretained through reversed-phase sorbents and can be effectively eliminated from the analyte in this manner. The procedure is as follows. The sample is loaded onto the SPE column in an aqueous buffer, then washed with dilute aqueous acid (e.g. 0.1% trifluoroacetic acid, TFA) to remove salts and low molecular weight contaminants. Peptide analytes of interest are eluted with a mixture of organic solvent (acetonitrile or propanol) in water containing 0.1% TFA. The SPE sorbents useful for peptide retention, in increasing order of hydrophobicity, can generally be stated as cyano < C2 < phenyl < cyclohexyl < C8 < C18. Very polar peptides should be isolated using sorbents with a high retention ability such as C8 or C18. Very hydrophobic peptides could be isolated with a less retentive sorbent such as cyano or C2. Medium and highly hydrophobic peptides could be efficiently isolated and fractionated with phenyl and cyclohexyl sorbents.

SPE based on ionic interaction of proteins can efficiently fractionate peptide mixtures into neutral, acidic and basic pools. In ion exchange chromatography, adsorption of proteins depends on the protein's isoelectric point relative to the column pH. Proteins with a high isoelectric point will bind tightly to a cation exchange column in the presence of a low pH and a low salt concentration. Proteins with a high isoelectric point will bind tightly to a cation exchange column in the presence of a low pH and a low salt concentration. Proteins with a low isoelectric point will bind tightly to an anion exchange column in the presence of a high pH and a low salt concentration. Hydrophobic interaction chromatography uses a high salt concentration to induce an interaction between hydrophobic regions of a protein and a weakly hydrophobic column packing. In all three cases, elution of the bound proteins can be achieved using a salt gradient.

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On-Line Preconcentration using SPE

The techniques of CE and on-line CE-MS have been widely documented for the analysis of therapeutically important peptides of diverse nature. A limitation of CE is that it works best for small sample volumes (typically < 50 nL for a 50 μm internal diameter capillary). This volume restriction leads to a poor concentration limit of detection (CLOD) when compared with typical HPLC and LC-MS systems. The incorporation of a membrane preconcentration cartridge (containing SPE in a membrane format) in-line with the CE capillary has allowed the introduction of much larger sample volumes (e.g. 100 μL), lowering the CLOD. Typical materials used for the preconcentration are reversed-phase sorbents, such as SDB and C18 bonded silica. This technique has allowed the analysis of biomolecules present in complex matrices, such as proteins in aqueous humour.

Pharmaceutical Applications

Bacitracin Extraction from a Pharmaceutical Ointment

Bacitracin ointment, an oily pharmaceutical formulation, is a mixture of at least nine antibiotic polypeptide complexes. These peptides are very polar and soluble in water and ethanol, but not in acetone or hexane. They can be separated from the ointment base by adding chloroform to the sample matrix, and the polar peptide antibiotics are adsorbed to a polar diol SPE column. Upon addition of the matrix to the column, the nonpolar solvent and ointment products pass through. A wash of chloroform removes potentially interfering components of the formulation. Antibiotics are removed from the sorbent using 0.1N HCl; protons from the acid displace the drugs from the hydroxyl groups on bonded silica surface. This technique can be useful for other drug substances by optimizing the SPE conditions for the properties of the drug and excipients, and selecting the appropriate sorbent and eluent systems.

Analysis of Aspirin Content in Tablets

Aspirin can be analysed for content in tablets by using a mixed mode sorbent containing both anion exchange and reversed-phase characteristics. Polysorb MP-2 (Interaction) polymer is a cross-linked vinylpyridine. At low pH the polymer is protonated and exhibits anion exchange and reversed-phase properties. At high pH, the polymer is neutralized and exhibits only reversed-phase properties. The sorbent is conditioned with acid/organic 10/90 (v/v) to induce polymer ionization. After sample loading, the sorbent is washed with 20–50% acetonitrile/water to neutralize the sorbent bed prior to elution of bound aspirin. Elution is accomplished with acetonitrile 30% NH_4OH–30 mM diammonium sulfate monohydrate (6 : 2 : 1, v/v/v). The polymer becomes neutral at this basic pH > 12.9 and aspirin remains ionized, disrupting its interaction with the sorbent and eluting. Salt acts as counteranion to further assist in the elution of aspirin.

Clinical Applications

Catecholamines from Plasma and Urine

Catecholamines (e.g. dopamine, epinephrine and norepinephrine) are of clinical interest for their role in neurochemistry as diagnostic indicators of phaeochromocytoma. These dihydroxylated amines are commonly analysed by HPLC with electrochemical detection. Many different SPE sorbents have been reported for their sample preparation. Alumina particles (about 50 mg) are added to plasma and buffer in a suspension, followed by centrifugation and subsequent elution from alumina. Reversed-phase C18 has also been used, as well as phenylboronic acid and strong cation exchange (SCX). The urine analysis of catecholamines examines metabolites such as vanillylmandelic acid and homovanillic acid. Typically, solid-phase extraction (SPE) uses SCX sorbent to provide cleaner chromatograms, either alone or in addition to alumina or phenylboronic acid.

Lipids

The extraction of lipids, including phospholipids, fatty acids, cholesterol, cholesteryl ester, and triglycerides in serum has been accomplished using polar SPE sorbents such as silica and aminopropyl. The sample matrix is extracted with a nonpolar solvent such as chloroform, and this extract is passed through a preconditioned polar aminopropyl sorbent for attraction of analytes by hydrogen bonding and weak ion exchange mechanisms. Neutral lipids are eluted with chloroform–propanol (2:1, v/v), fatty acids are eluted with 2% acetic acid in diethyl ether, and phospholipids are eluted with methanol. The neutral lipid fraction is evaporated and reconstituted in hexane. The hexane mixture is then passed through a second amino SPE column. The cholesteryl esters are eluted with hexane, with the second column in series with the first column to trap cholesterol, which elutes with triglycerides. Triglycerides are eluted with hexane–diethyl ether–methylene chloride (89 : 1 : 11, v/v). The two amino columns are separated and cholesterol is eluted from both – di- and mono-glycerides elute from the upper amino column. Cholesterol is eluted with 5% ethyl acetate in hexane, diglycerides are eluted with 15% ethyl acetate in hexane and monoglycerides are eluted with chloroform–methanol (2 : 1, v/v).

Drugs in Tissues

The majority of reported methods for drug extraction involve plasma, serum, urine or other polar fluids that can easily pass through SPE cartridges. However, blood and tissue extraction applications have not been reported with much frequency in the published literature. The extraction of drugs from tissues, such as liver, kidney, intestine, brain, muscle and adipose tissue is important for the forensic toxicologist, in particular, since urine and blood are not available in post-mortem cases. Concentrations of drugs in tissues are also of great interest to researchers investigating the deposition of drugs in certain tissues (e.g. ophthalmic drug delivery into the eye and delivery of antidepressant drugs into the brain).

Drug extraction from tissues involves first homogenization of the tissue with aqueous solution. After homogenization, an enzyme digestion (e.g. Carlsberg subtilisin, lipase or protease) step and/or protein precipitation can be used, followed by centrifugation. Sometimes a small percentage (10–20%) of organic solvent (e.g. methanol) is added to the water or buffer for homogenization, and the solution is passed through an SPE cartridge. Alternatively, 100% methanol or acetonitrile can be used and, after centrifugation, the solvent is evaporated, reconstituted in aqueous solution or buffer, and passed through an SPE cartridge. In addition to typical SPE sorbents, diatomaceous earth is another choice for the analyst; it facilitates a liquid–liquid extraction by attracting analytes to the particles to increase surface area available for extraction when an organic solvent is passed through the diatomaceous earth column. The use of 'high-flow' SPE columns is now a reality owing to larger particle-size sorbents in columns, typically 100–120 μm particle sizes instead of 40–60 μm.

Conclusion

Solid-phase extraction has been demonstrated to be a reliable and cost-effective technique for the selective isolation and concentration of a wide range of analytes and sample matrices, and offers many improvements over traditional techniques such as liquid–liquid extraction. Some of the classic applications for SPE include environmental trace enrichment of organic pollutants, extraction of pesticides and growth promoters from foods, purification of peptides, drug analysis in pharmaceutical dosage forms and clinical applications for drugs in physiological matrices. Its ability to solve sample preparation problems has been well documented in the literature over the past two decades. There is a wide choice of sorbents for SPE, including nonpolar, polar, ion exchange and mixed mode chemistries, providing the analyst with the selectivity necessary to obtain clean extracts for analysis. SPE can be used either manually or with greater throughput using automated workstations. The introduction of new sorbents with more selective modes of attraction, novel product formats

such as the SPE disc, and the proliferation of automated techniques for performing the extractions, ensure that SPE will continue to be a preferred technique for sample preparation in many different analytical disciplines.

See also: **II/Extraction:** Solid-Phase Extraction. **III/Solid-Phase Extraction with Discs**.

Further Reading

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SOLID-PHASE EXTRACTION WITH DISCS

C. F. Poole, Wayne State University, Detroit, MI, USA

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Introduction

Solid-phase extraction is a well-established technique for the isolation, concentration and matrix simplification of analytes in samples with unfavorable properties for direct analysis by the best available approach. Extraction is achieved using a particulate sorbent packed into columns of short length (sometimes called 'cartridges') or immobilized in the form of a thin disc, referred to generically as 'disc technology'. Since the same sorbent chemistry is used for the extraction step and liquid desorption for the elution step in both approaches, the two techniques differ only in format. On an evolutionary scale, solid-phase extraction using short columns was introduced as a laboratory-scale technique in the late 1970s and came to prominence in the 1980s. Disc technology, by comparison, was first introduced in 1989, and is still evolving as a competitive technique to short packed columns. Simply stated, disc technology should be viewed as an alternative approach to performing solid-phase extraction with additional benefits and capabilities derived from the difference in format. Whereas packed columns are easily prepared in the laboratory for evaluating new sorbent chemistries and evaluating sampling properties, discs, so far, have only been produced in a manufacturing setting. Consequently, sorbent selection and device optimization have been restricted by market-driven considerations. As a consequence, the main applications of disc technology are generally narrowly focused on the needs of large volume users more so than is the case for conventional short packed columns.

Disc Formats

Solid-phase extraction discs are available in different styles and sizes. Particle-loaded membranes (Empore™ discs) contain 8–12 μm sorbent particles homogeneously distributed in a web of short poly (tetrafluoroethylene) (PTFE) fibrils. These are formed into 0.5-mm thick discs with diameters from 4 to 96 mm. They are flexible and superficially resemble filter paper discs. They are used with some supporting structure such as a frited glass filter or porous plastic support. The discs contain about 90% by weight of sorbent with the balance being the PTFE microfibrils. Some characteristic physical properties are indicated in **Table 1**. Particle-loaded membranes are also available in a syringe barrel format similar to conventional short packed column-sampling devices. In this case, the sorbent bed contains particles of a larger diameter, about 50 μm, in thicker discs, about 1.0 mm, sealed into the base of a 4 mm (1 mL), 7 mm (3 mL), 10 mm (6 mL) and 20 mm diameter (40 mL) open syringe barrel. These discs have an integral prefilter consisting of a graded density of poly(propylene)